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PROPERTIES OF MATERIALS USING ACOUSTIC WAVES.(U)
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OFFICE OF NAVAL RESEARCH
CONTRACT NO. N00014-76-C-0527

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SUMMARY REPORT

covering

Nov. 1975 - Present

R.E. Apfel, Principal Investigator

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I INTRODUCTION

This is a Summary Report covering the activities supported under ONR Contract N00014-76-C-0527 for a period of six and one half years, from 1 November 1975 to the present (1 May 1982). During this period ONR has funded research in physical acoustics and in the properties of materials (including biological ones) and their phase transitions at an average annual rate of approximately \$43,500, for a total of approximately \$282,000 (which includes a prorated portion for the current contract year). This amount has supported the research of thirteen individuals through salaries, equipment and materials, travel, publications, etc.

In this report we shall summarize in Section II the principal research thrusts of our program. Rather than a chronological study, which can be found in past Progress Reports and Annual Summaries, we shall summarize our accomplishments in each of several areas. Spin-offs to our research efforts are covered in Section III. In Section IV we offer some concluding remarks, in Section V we list publications related to the objectives of the contract, and in Section VI we list the personal involved on the work of the contract.



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II FOCUSES OF OUR RESEARCH EFFORTS

Our research efforts can be classified into four broad categories; Applications of acoustic levitation; nonlinear acoustics and radiation pressure; acoustic cavitation; and the development of a facility for the characterization of small samples. Although these classifications are somewhat arbitrary, with major overlaps existing among different categories, they do allow us to discuss the major thrusts of our work.

A. Applications of Acoustic Levitation

When the acoustic radiation stress on an object produces a force that balances the gravitational force on it, the object will be levitated. One can either use levitation to characterize the sample mechanically, in a manner analogous to Millikin's characterization of the electric charge on an oil drop, or one can merely use the levitation as a tool for manipulating the sample.

1. Bulk Characterization; The Compressadensity

Our earliest work (Harvard Ph.D. thesis) employed acoustic levitation to measure the tensile strength of liquids. Since acoustic levitation of a sample depends on differences in the density and compressibility of the sample, as compared to the host liquid in which the sample is levitated, we soon realized that we could deduce from the position of the levitated sample a simple factor that depended on the density difference and the compressibility difference. We called this factor the compressadensity and measured it for several liquids of known properties; we found that deductions of the sound velocity from the compressadensity for submicroliter samples were within 1-2% of the results using standard

laboratory techniques that usually require in excess of 30 milliliters.

The next question was, for what kinds of samples is this technique particularly useful? Four classes of materials fall into this category: metastable liquids (superheated, tensilely stressed, or supercooled) which are difficult to maintain in this fragile state in large quantity or in contact with solid materials; rare materials, such as pure biological materials, which are sometimes not available in quantities more than a fraction of a gram; dangerous or toxic liquids which you desire to characterize; and materials whose properties depend on their host environment; for example biological cells, which have properties that depend on osmosis. (See Section III).

We have calculated from levitation data the sound velocities of n-pentane to 140°C (105° above its boiling point at atmospheric pressure) and hexane to 170°C (101°C above its boiling point). Even more interesting are levitation tests of the properties of supercooled water. Our results have shown a new anomaly for liquid water: a turning point in the sound velocity at approximately -33°C. These are the first of only a few bits of data that argue against the more popular speculation that water is approaching (but never reaching) a critical type of behavior at around -45°C. The results are certainly relevant to the fundamental understanding of the structure of water; moreover, the results have relevance to the question of what is the likely mode of solidification of supercooled water — a question of concern to cryobiologists.

We have also measured the properties of several lipid oils (only available in less than gram quantities) to answer questions such as: Can the porpoise adjust the relative proportion of lipid isomers in its

dome, thereby altering the acoustic properties of this acoustic lens?

Our data does not support this hypothesis.

2. Surface Characterization

The natural frequency at which a drop of a liquid in another liquid undergoes quadrupole shape oscillations is a key to the interfacial tension associated with the liquid-liquid interface. In order to measure interfacial tension in this way, it is convenient first to levitate the sample and then to drive it into shape oscillations. The theory for forced shape oscillations of a liquid drop on another liquid was carried out first by Marston in our lab. Exciting shape oscillations is difficult experimentally because acoustic wavelengths at the natural frequency for shape oscillations are much greater than the drop diameter. We overcame this problem by using acoustically generated radiation stresses at a high acoustic frequency to produce a static deformation of the drop, and then modulating (i.e. turning on and off) this deformation at a much lower rate, looking for the modulation frequency which defined the resonance condition. Since deformations are on the order of microns, we have used different optical techniques to detect them. In *rainbow interferometry* coherent light rays that reflect and that refract and internally reflect will, upon leaving the drop, interfere with each leading to an interference pattern. That pattern is blurred when the drop is resonating in its quadrupole mode. In *rainbow photometry* light that scatters at an angle normally associated with the rainbow is compared in phase with the driving modulated stress on the drop to allow for the prediction of the resonance condition. In instances when the indexes of refraction of the two liquids are not appropriate to rainbow

photometry we have used a *shadow imaging* technique developed by Trinh. Currently Marston at Washington State, Trinh at the Jet Propulsion Laboratory, and Hsu, Zhu and I at Yale are using this *modulated radiation pressure* technique. We are currently measuring the interfacial tension of various superheated hydrocarbons with water; (propane, butane, et al). Our results are inconsistent with the theory of Fowkes which many researchers are using. These tests outside the normally accessible range are, therefore, indicative that the present understanding of forces at a liquid-liquid interface is in need of revision.

The use of modulated radiation pressure to study the deformability of cells is discussed in Section III.

3. Transport Properties

The modulated radiation pressure technique can also be used to deduce the viscosity of the sample. This possibility exists because we can measure the relative phase between the excitation signal and the movement of the drop surface for several different modulation frequencies. Current comparisons of the viscosity measured in this way with literature values are imperfect, varying by as much as 50%, suggesting that Marston's model for the damping is in need of revision or that surface contamination must be taken into consideration. If the discrepancy can be sorted out, then this relative phase technique may be a new and valuable tool for characterization of surfaces in the presence of contaminants.

4. Manipulation and Separation using Acoustic Levitation

One of the most obvious uses of acoustic levitation is in manipulation of the sample. In our earlier work we used a levitated sample to study with high speed movies the vapor explosions of superheated drops. The Jet Propulsion Laboratory group headed by Taylor Wang has taken the manipulation aspects of acoustic levitation to the extreme preparing modules for the Space Shuttle that will perform tests ranging from basic fluid mechanics experiments to the solidification of molten materials in the absence of a solid container.

One of the most useful features of acoustic levitation is that the position at which a drop is levitated is independent of its size if the acoustic wavelength is large compared to drop diameter. That is, small and large drops of that material will all go to the same position in the acoustic field. This implies that a levitation cell could be used for the spatial separation of different materials. In a vertical acoustic standing wave field characterized by alternating series of acoustic pressure minima and maxima, we could have the following four way separation. Samples that are more compressible and less dense than the host liquid would migrate to positions just above a pressure maxima; those more compressible and more dense would drop below the maxima. The less compressible samples would migrate to the pressure minima, with less dense samples being above and more dense samples being below. The crux of such a separation is, of course, the choice of the appropriate host material for a given separation.

More on separation and manipulation is discussed in Section III.

B. Nonlinear Acoustics

Two major aspects of our program in nonlinear acoustics are 1) the theoretical study of the acoustic force on an object, whether in the one dimensional or three dimensional case and 2) the study of nonlinear propagation in liquids and tissues, with possible applications in medicine.

1. Fundamentals of the Theory for the Acoustic Force on an Object.

We have divided this subject into the case that is relevant to acoustic levitation of a liquid or biological sample, in which the non-linearity parameter of the sample is not relevant, and the one dimensional plane wave case, in which the nonlinearity parameter is important if the column is laterally constrained.

In the three dimensional case there are a variety of questions which are relevant to different situations. For instance, viscosity is not important in the theory for the levitation of 1 mm diameter samples at 50 kHz. For 1 μ m samples at the same frequency the viscous boundary layer is comparable to the radius of the sample and therefore cannot be neglected. Shape can have an effect if the sample is sufficiently more or less dense than the host medium. Interparticle forces between neighboring particles, due to secondary radiated fields, can become important especially for large and/or compressible samples and samples that are within a few diameters of each other. Currently, these effects are accounted for by using an *ad hoc* approach which corrects existing theory for inviscid liquids and spherical samples. We are currently trying to approach some of these problems from first principles.

We (Prof. Chu predominantly) have been preparing a tome, which is

currently in the final stages of the review process, on the subject: Acoustic radiation pressure produced by a beam of sound. One might expect that the one dimensional case of nonlinear propagation in an inviscid fluid might have been solved by now, yet a controversy has raged on this subject from the time of Rayleigh to the present time. We believe that our critical review of prior work coupled with a new approach based on the fundamental conservation principles will clarify the particular case of the acoustic force on a target for a laterally confined beam and will offer a more general framework for other nonlinear problems.

2. Nonlinear propagation in liquids and biological materials .

In many situations the nonlinear parameter of a liquid (or third order elastic constants of a solid) is measured by using a disc transducer and a similar receiver, with the second harmonic of the transmitted signal being indicative of nonlinearities in the momentum equation and in the properties of the material. Most approaches either use a plane wave theory or a theory that corrects for some diffraction effects of the finite piston transducer. Not only must diffraction be included to second order but dissipation must be included in any theory which is to be used with experimental data on the second harmonic if an accurate nonlinear parameter is to be deduced. An appropriate dissipationless theory has been corrected for dissipation by Cobb for use with his data on harmonic generation in liquids, biological solutions, and tissues. Two major questions can be resolved by such measurements: Can the nonlinear parameter provide a useful indication of

pathology in tissues? And can the second harmonic information be used to improve the images in diagnostic instrumentation. Initial indications from Cobb's Ph.D. thesis are that the nonlinear parameter does *not* vary widely for a wide range of tissue parameters; the linear absorption of sound may be a better indicator. Improvement in diagnostic imaging will be one of the topics Cobb investigates in his year as Hunt Postdoctoral Fellow (an honor he received from the Acoustical Society of America).

Cobb was also able to demonstrate in his thesis the effects of tissue aging and phase cancellation on measurements of linear and nonlinear properties; he also adopted procedures for calibration of biomedical transducers which should be useful in the medical field.

C. Acoustic Cavitation

When asked by Peter Edmonds to write a chapter on acoustic cavitation for a volume on Ultrasonics for the renown series *Methods of Experimental Physics*, my first thought was that such a task could take an inordinate amount of time. I was right! But I took the task, partly out of egotism and partly because I felt the existing well-known reviews on this topic were authored by theorists who usually concentrated on the solutions of the equations that attempt to model the dynamics of a bubble in an acoustic field. Acoustic cavitation includes much more than this. It includes gas and vapor bubble nucleation; and it includes a consideration of the practical aspects of either minimizing or optimizing the effects of cavitation. Moreover, cavitation — like cancer — is a generic term that covers phenomena that are controlled by different mechanisms.

My earlier work had considered the aspects of nucleation that relate to the tensile strength of liquids; and in separate work I considered the role of solid, imperfectly wetted impurities which greatly lower the maximum tension a liquid can sustain.

The chapter on acoustic cavitation took longer than expected because I would not resist the temptation to try to fill in some of the missing pieces of the puzzle. For instance, the prediction based on numerical analysis of two resonances for vapor bubbles was put on a firm physical basis by Marston while he was in our laboratory; this new interpretation of an evaporation-condensation resonance was included in the chapter and also published separately by Marston. The dynamics of bubbles in high intensity sound fields are often seen for air bubbles in water. In the chapter, we included nondimensionalized bubble radius vs time plots for a whole range of pressure amplitudes, acoustic periods, and for a range of viscosities and surface tensions.

Our most important new contribution was a new method for estimating a "transient cavitation threshold" for a sample. This new prediction was combined with thresholds for rectified diffusion and for gas bubble nucleation to give a series of cavitation prediction charts that allows the user to estimate the type of acoustic cavitation he is likely to encounter for a given situation (i.e. for a given frequency, acoustic pressure, gas saturation, and estimated bubble nuclei distribution).

Several, including P. Marston and E.A. Neppiras, helped a great deal in the preparation of the manuscript. (Neppiras has also recently published in Physics Reports an excellent review that concentrates on the

work in bubble dynamics.)

These cavitation prediction charts have also permitted us, in a recent paper, to make estimates of potential bioeffects from diagnostic ultrasound. One conclusion that must be taken seriously: we show that at current intensities of diagnostic equipment, a bubble of a fraction of a micron radius can grow in an aqueous environment during a single acoustic cycle to a radius of the order of 5 μm and then collapse into a region of smaller than cell dimensions, depositing about 300 MeV of energy. We have put our energy in the units used by those who estimate damage from ionizing radiation to dramatize the reason for our concern. Our predictions do not prove that a problem exists, but suggests that further research is warranted and that diagnostic sound may not be sound as a *routine* procedure for pregnant women.

D. Small Sample Facility

Our acoustic levitation facilities are complemented by commercial equipment to facilitate the measurement of mechanical bulk, surface, and transport properties of materials (predominantly liquid samples). Our capabilities are summarized below.

<u>Property</u>	<u>Type of Apparatus</u>	<u>Minimum Sample Size</u>	<u>Expected Accuracy</u>
Density	Mettler Density Meter	0.1 cm^3	<0.01%
Sound velocity of stable liquids	Acoustic levitation	0.1 μl	1-2%
Sound velocity of metastable liquids	Acoustic levitation	0.1 μl	2-3%
Sound velocity of stable liquids	Commercial sing-around velocimeter	30 ml	<0.02%

Interfacial tension	Modulated Radiation Pressure of Acoustically levitated samples	1 ml	3-5%
viscosity	Modulated Radiation Pressure of Acoustically levitated samples	1 μ l	uncertain at this time

Temperature control for the commercial apparatus is usually within $\pm 0.05^{\circ}\text{C}$, whereas our control in the levitation cells is $\pm 0.5^{\circ}\text{C}$.

III SPIN-OFFS

Several of the phenomena studied and the techniques developed originally under ONR support have been transferred either to specialized research projects for which we receive support from other agencies or to other laboratories. Some of these projects are outlined below

A. Neutron Detector

Superheated drops are sensitive to radiation, as Glaser showed with his invention of the bubble chamber in 1952. We intended to acoustically levitate single superheated drops to find the conditions under which neutrons of a given energy would trigger droplet vaporization. I realized in 1976 that many drops could be immobilized by a gel, rather than via acoustic levitation. Without the need for electronic apparatus the device became a pocket neutron dosimeter for which I received a U.S. Patent in 1979. NSF supported the research from 1975-1980, (the U.S. Government has rights in my patent) and D.O.E. currently supports my research in this area.

B. Characterization of Biological Cells with Acoustic Waves

A number of current projects deal with the interaction of ultrasonic waves with biological tissues. Some of these are supported by the National Institutes of Health. In particular, we have used acoustic levitation to characterize blood components (red blood cells, lymphocytes, and platelets). We are also looking at the scattering of high frequency (25 MHz) tone bursts from single cells so that we can characterize populations of cells.

C. Cell Deformation and Separation

We would like to use the modulated radiation pressure technique to study the deformability and shape resonance of single red blood cells while they are being observed under an optical microscope. The frequency of resonance should be a good indication of the surface and intracellular viscosity, and therefore should be useful for studying diseased states (e.g. sickled cells).

We are also interested in biological cell separation using the levitation technique, as discussed in Section II. A commercially practical separation technique of this sort would be the first ever that would depend in part on differences in the elastic properties of materials.

D. Work at Other Universities

P. Marston at Washington State University, E. Trinh at the Jet Propulsion Laboratory and U. Varanasi at Seattle University are all using acoustic levitation and/or modulated radiation pressure techniques developed in our laboratory at Yale. Researchers at San Francisco State and Brookhaven National Laboratory are currently exploring our radiation detection work. The healthy interchange of data and sharing of techniques has been extremely useful to each of us in unraveling the puzzles on which each of us chooses to work.

It certainly should be mentioned that the work of ours at Yale is itself a spin-off from my earlier activities in Harvard's Acoustic Research Laboratory directed by F.V. Hunt and supported generously by ONR.

IV CONCLUDING REMARKS

The work supported by ONR under this contract, though fundamental in many respects, finds application in many areas. For instance, the whole subject of using intense acoustic waves to exert forces on particles (e.g. to separate, manipulate, and/or coalesce them, etc.) opens up a new set of applications of macrosonics for industry and medicine. A fundamental understanding of acoustic cavitation phenomena offers opportunities for applications ranging from sonar systems to the consideration of bioeffects due to diagnostic ultrasound. And the subject of nonlinear acoustics interfaces with the broader area of fluid mechanics, with its many relevant problems.

Our team effort at Yale has spawned related efforts elsewhere, with Marston building a strong acoustics program at Washington State, Trinh working on space applications of macrosonics at the Jet Propulsion Laboratory, and Weiser carrying her acoustical expertise to the Exxon Production Research Co. in Houston.

We look forward to continuing our work in nonlinear acoustics, macrosonics, and material characterization. It is usually not clear in what areas the breakthroughs will come. By offering a broad program of graduate study in acoustics, we hope to provide an atmosphere in which such breakthroughs are more likely. With that in mind we shall, in the near future, be petitioning the Executive Committee of the Faculty of Arts and Sciences of Yale University to establish a new Center which will emphasize research in ultrasonics (the name hasn't been chosen yet). This Center would be a confederation of Yale researchers from the areas of

physical acoustics, underwater acoustics, signal processing, imaging, and medical ultrasound, who would share their research experiences and collaborate on subjects of mutual interest in the basic and applied sciences.

V PUBLICATIONS UNDER N00014-76-C-0527 from 11/75 - 4/82.

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M.A. Weiser and R.E. Apfel, "Extension of Acoustic Levitation to Include the Study of Micron-size Particles in a More Compressible Host Liquid," J. Acoust. Soc. Am., in press.

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Technical Memorandum No.2, E.H. Trinh, Nov. 1977

THE PROPERTIES AND STRUCTURE OF LIQUID WATER: AN OVERVIEW.

Technical Memorandum No.3, E.H. Trinh, Nov. 1977

THE SOUND VELOCITY IN SUPERCOOLED AND SUPERHEATED WATER UNDER ATMOSPHERIC PRESSURE.

VI. PERSONNEL WHO HAVE WORKED ON PROJECTS SUPPORTED BY N00014-76-C-0527*

<u>NAME</u>	<u>TITLE</u>	<u>TIME ON PROJECT</u>
Robert Apfel	Principal Investigator	11/76 - present
K. Baxter	Undergraduate student and part-time summer employee	During term and summer, 1977
B.-T. Chu	Professor	1979 - present
Howard Clifton	Summer Res. Program Participant	Summer 1978
Wesley Cobb	Graduate student	1978-1982
Chaur-Jian Hsu	Graduate student	1977-1982
P. Marston	Research Fellow	1976-1978
E. Neppiras	Consultant	1 week each Fall
E. Trinh	Graduate student Postdoctoral Res. Assoc.	1972-1978
Victoria Wagner	Summer Res. Program Participant	Summer 1977
M.A. Weiser	Graduate student	1976-1981
Roberta Young	Summer Res. Program Participant	Summer 1976
<u>Zhu</u> Zhe Ming	Visiting Fellow	1981 - present

* Often support has also come from other sources (e.g. Yale); these individuals have in one way or other supported the objectives of the contract.

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